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USING PHASE TRANSITIONS TO IMPROVE THERMOSTATIC CONTROL OF INSTRUMENTS

by K. Ye. Veselov, L. V. Kalisheva, and M. L. Telepin

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USING PHASE TRANSITIONS TO IMPROVE THERMOSTATIC CONTROL OF INSTRUMENTS

K. Ye. Veselov, L. V. Kalisheva and M. L. Telepin

Existing methods for the thermostatic control of gravity meters and their shortcomings are discussed and the use of phase transformation for the accurate control of temperature is proposed. Experimental investigations carried out for the purpose of developing phase transformation thermostats since 1954 are discussed. Experience in the use of ice, gallium and its eutectic alloys, and saturated hydrocarbons in phase transformation thermostats is reviewed.

Methods of Thermostatic Control and Their Shortcomings

When particularly fine physical experiments are conducted, the devices are /145* thermostatically controlled by placing them into the Dewar flask. An example of such a physical experiment is the observation of the force of gravity when using a gravity meter which, for purposes of geological prospecting, must pro-

vide a relative accuracy of the order of 10^{-8} . For such stringent requirements of accuracy, even the Dewar vessel does not provide the necessary thermal insulation. It has been shown in practice that on days when the temperature of the external air varies sharply, high accuracy cannot be expected from field observations. It is true that in recent years an improved thermostatic control of gravity meters has been developed at the All-Union Scientific Research Institute of Geophysics. However, this technique works well only under conditions of a stationary thermal state. The three-dimensional spread of the sensor and of the compensator, complicated by the presence of intermediate mechanical devices, which are in a state of stress, serves as the reason for errors in the device when there is the slightest temperature heterogeneity in it. Therefore, temperature change, as before, remains one of the basic causes inhibiting highly accurate measurements by means of a gravity meter.

Let us consider the methods which can be used to improve thermostatic control. On the one hand we should improve the thermal insulating properties of the Dewar flask. However, a technical limit has already been achieved in this direction and it is very difficult to surpass this limit. On the other hand we can concern ourselves with the improvement of the thermal stabilizing action of the substance which is inside the Dewar flask. In this direction there are still possibilities which have not been exhausted.

A simple increase in the mass contained within the Dewar flask (for the purpose of increasing heat content) is not sufficiently effective even though it smooths out the external temperature fluctuations. The shortcomings of this

*Numbers given in margin indicate pagination in original foreign text.

approach are that by increasing heat capacity we achieve only passive thermostatic control: by absorbing the heat flux flowing into the Dewar flask the heat absorbing mass inevitably changes its temperature. Some constructions incorporate the active or forced thermostatic control in the Dewar flask by introducing an electrical heating element which is turned on by a regulator as soon as the sensor feels that the temperature has dropped below the acceptable point. However, this method too is associated with serious defects: at the present time in the world's literature there is no agreement on a substantial increase in the accuracy of quartz stabilized gravity meters when an electrical thermostat is used. Apparently this is explained by the imperfect reaction of the thermostat servosystem and by the high sensitivity of a gravity meter to a change from a heating mode to a cooling mode. The shortcomings of an electric thermostat also include the complexity of the device, its dependence on the power sources and the necessity of artificially moving into the region of high temperature thermostatic control, substantially in excess of external temperatures, because only in this case does the thermostat operate with stability. Methods utilizing variable heating and forced cooling are even more complex and their applications to large thermostatically controlled volumes are completely undeveloped. /146

Two-Phase Thermostatic Control

We have carried out investigations on the utilization of low melting substances during their phase transformation from solid to liquid state and vice versa for use in thermostatic control during superaccurate gravimetric measurements. The counterreaction to changes of temperature in this case is achieved by using the latent energy of phase transformations (fusion-solidification) of low melting substances. A thermostat operating on this principle combines the best properties of thermostats which have already been considered.

On the one hand we have a ready reaction to changes of external temperature. The energy effect of phase transformations is much more pronounced than the action of the same mass working as a simple heat capacity. Thus, if one calorie (according to our investigations, this quantity of heat flows into the Dewar flask during a period of ten hours when the temperature differential is ten degrees) is absorbed by a liter of water, its temperature will still rise one degree while the same liter of water transforming from a solid state into a liquid state is capable of absorbing 84 kilocalories and does not undergo any temperature change.

On the other hand the reaction of the above two-phase thermostat to temperature deviations is based on molecular processes whose mechanism of action, as a rule, takes place without failure (if the phenomenon of supercooling does not take place as discussed below). At the same time the feedback network from the temperature deviation to the liberation of the necessary amount of energy, is reduced to a minimum and the possibility of a delay in the reaction to this deviation is also reduced to a minimum. In this respect this method resembles passive thermostatic control.

Let us compare the energy capacity of dry cells which power the electric /147

thermostat and the capacity of the active material of a two-phase thermostat. A dry cell weighing 650 g produces a current of 0.12 amp and with an average voltage of 1.2 V during a period of 250 hr, liberates 0.144 Joules/sec·250·3600 sec=130 kiloJoules of energy. On the other hand 650 g of water, while freezing, liberate 650·84=54.6 kilocal=238 kiloJoules. As we can see, the advantage of water is obvious.

In the case of gallium or hydrocarbon, the energy effect is less pronounced. Their heat of fusion is from 10 to 30 cal/g. Therefore, 650 g of the substance liberates from 27 to 81 kiloJoules. However, this amount is not too small, particularly if we remember that an electric thermostat operates with a larger difference between external and internal temperature while in the case of a two-phase thermostat this difference can be reduced to a minimum. Furthermore, in the process of external temperature fluctuations which becomes greater or less than the melting point of an active substance, the substance is either melting and absorbing thermal energy or solidifying and liberating thermal energy, thus retaining its capacity to do useful work for a long period of time. In other words a two-phase thermostat is reversible with respect to energy flow whereas an electric thermostat can only liberate thermal energy.

Finally, the unique advantage of a two-phase thermostat over the electric thermostat is its capacity to compensate for "thermal shocks" when it becomes necessary to develop thermal fluxes of tremendous power for short periods of time. In this respect a combination of an electric thermostat with a two-phase thermostat is very enticing. In this case the two-phase thermostat will be capable of smoothing out and correcting all of the occurring errors and roughnesses in the control of the thermal balance.

Let us consider the principle, which we have described, in action. Figure 1a has been constructed from the results of many repeated experiments and is confirmed by theoretical calculations. Ten grams of water are placed in a Dewar flask. This thermostat, which is initially at room temperature, is placed in an environment of minus ten degrees. Before the water starts to freeze it works like a simple heat capacity. The variation in the temperature, T , inside the Dewar flask with time is proportional to the difference in the temperature inside and outside ($T - T_{out}$), is inversely proportional to the total

heat capacity C and has a negative sign because it decreases this differential

$$\frac{\partial T}{\partial t} = -\frac{H}{C}(T - T_{out}),$$

where t is time; H is the heat flux flowing into the Dewar flask during a standard differential; C is the total heat capacity of the Dewar flask contents.

According to our investigations, for conventional vessels which are not defective $H=10$ cal/hr degree. Integrating this differential equation we obtain an exponential law for the variation in temperature with time:

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$$T - T_{out} = (T_0 - T_{out}) e^{-\frac{H}{C}(t - t_0)},$$

where T_0 is the temperature inside the flask at the initial instant of time t_0 .

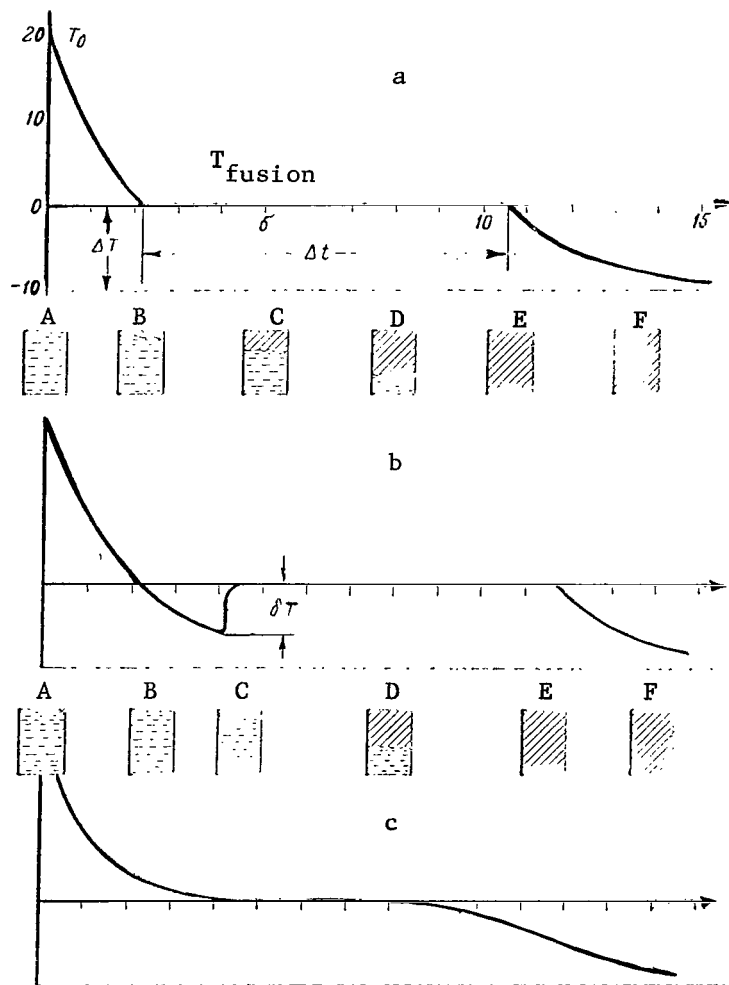


Figure 1. Generalized graphs for the cooling of a substance undergoing phase transformation. a, in the absence of supercooling; b, in the presence of supercooling; c, in the case when the substance contains impurities.

In our example $T_{\text{out}} = -10$ degrees, the initial instant of time is taken as 0 /149
 $(t_0=0)$ the corresponding initial temperature is $T_0=20^\circ$, the total heat capacity of water and of the internal flask walls is equal to 20 cal/degree. Consequently, the temperature curve is given by the equation

$$T = -10^\circ + 30^\circ e^{-\frac{1}{2}t},$$

where t is expressed in hours.

Such is the initial part of the temperature graph (the region AB). As the

water begins to freeze, temperature change is stopped. The heat flux (in our case it is negative, i.e., it is a flux of cold), which is entering the flask due to a temperature differential is now used up entirely for the phase transformation associated with the freezing of water. The quantity of liquid water decreases continuously while the quantity of ice increases correspondingly. The BCDE region of the temperature graph has the form of a horizontal line segment. In order to find its length we must divide the supply of latent fusion energy (the product of specific fusion heat q by the mass m of the active substance) by the heat flux during the specified temperature difference:

$$\Delta t = \frac{qm}{H\Delta T}, \Delta T = T_{\text{fusion}} - T_{\text{outside}}. \quad (1)$$

In our example $q=84$ cal/g, $m=10$ g, $H=10$ cal/hr·degree, $\Delta T=0-(-10^\circ)=10^\circ$, from which

$$\Delta t = \frac{84 \cdot 10}{10 \cdot 10} = 8.4 \text{ hr.}$$

Thus 10 g of water would be sufficient to maintain a constant temperature for a period of 8.4 hours. After this period has elapsed, the active substance --water--will deplete its energy capacity and will transform completely into a solid state, thereby reviving the exponential temperature drop (along the region EF of the graph) and will approach the external temperature asymptotically.

In order to bring the active substance back into proper operating state it must be heated until it is completely melted. In this case the active substance will continue to "operate" and will now absorb heat, producing a similar horizontal area on the temperature rise graph whose length is given by equation (1).

Figure 1b shows the curve for the case when the supercooling phenomenon is present: this phenomenon is caused by the fact that a particular molecular mechanism "does not operate" and the temperature of the substance continues to drop below the standard point of crystallization by an amount δt (supercooling interval), and only then intense crystallization (point C) takes place. In this case the temperature rapidly rises to the standard level of crystallization and is retained there until solidification is completed. Obviously, supercooling reduces the quality of thermal stabilization but in practice it is encountered only in some substances (gallium) and even there the temperature interval δt may be reduced to 0.5° or less by introducing special impurities and also by avoiding excessive overheating.

Another characteristic distortion of the temperature graph is shown in figure 1c. It is due to the presence of impurities and to the contamination of the active substance. As a rule impurities lower the solidification temperature of the basic material although the initial portions of the impurities may begin to precipitate from the solution at a relatively high temperature. It is important to note that each component crystallizes independently from the mixture forming individual grains in accordance with its own law. The composition of the remaining liquid phase changes during the process of crystallization.

At the same time the feasibilities of crystallization change: new portions of material transform into the solid phase only if there is a further decrease in temperature. In the end a mixture remains which is very difficult to solidify. As a result of all this we have an inclined region on the graph in place of a horizontal one and only a retardation in the temperature change rather than its stoppage.

It is also possible to have mixtures which do not change the composition of the liquid phase as they solidify. Such mixtures are called eutectics. From the standpoint of the temperature graph eutectics are identical to a pure substance which has a lower fusion point than each of its components.

Ice Thermostat

In 1954 an ice thermostat was applied to the quartz astatizing of a gravity meter in the gravimetric laboratory of the All-Union Scientific Research Institute for Geophysics. Its exploitation indicated that it was inadequate but, nevertheless, some of the shortcomings became clear. After an intense shaking of the device which had remained at rest, a discontinuity in the reading was observed. Apparently the reason for this is that the temperature of the various regions of the active material as well as the temperature of the walls adjoining the case is nonhomogeneous and when mixing takes place there is a sudden contact between the base and a substance with a different temperature, i.e., there is a thermal shock. Generally speaking, temperature heterogeneity is unavoidable because thermal fluxes exist. Temperature stratification is also produced because the cold parts of the substance, particularly ice, are constantly tending to float up.

The stabilization of the temperature state in order to make it independent of episodic shaking may be achieved in two ways: the substance can be mixed continuously so that its state will not change appreciably due to shaking, or, on the contrary, we may inhibit mixing by dividing the volume into individual parts and completely fill the air gaps. The situation is substantially improved if a heavy multilayer copper screen is distributed over the volume occupied by the active substance.

Another shortcoming involving the sweating of some glasses of the gravity /151 meter, indicates that the ice thermostat must be used during the cold part of the year, i.e., primarily in the solidification mode. This mode is also to be preferred from the standpoint of simplicity in obtaining heat under field conditions.

Thermostat Utilizing Gallium and Its Alloys

At the beginning of our investigations our attention was invited by low-melting metals--gallium and its eutectic alloys. Their advantage over ice is that by selecting a particular alloy it is possible to obtain different temperatures which can be thermostatically controlled. Thus pure gallium melts at 30°C, its eutectic alloy with aluminum melts at 26.5°, with zinc at 25°, with

tin at 20°, and with indium at 16.5°. The triple eutectic gallium-aluminum-zinc melts at 21.9°, the tetradic eutectic gallium-indium-tin-zinc melts at 7°. The selection of a material with a definite fusion temperature is significant because if it turns out to be close to the average diurnal temperature, then during the process of actual temperature fluctuation the substance operates alternately in the heating and cooling mode and remains in its active state for a prolonged period of time without depleting its capacity.

The specific heat of fusion for gallium and its alloys is 10 cal/g, i.e., 8 times less than for ice. However, since its density is also higher (6 g/cm³) the volume generating the necessary amount of heat in the case of gallium is approximately the same as in the case of water.

Among the special features of gallium and its alloys we should mention its thermal conductivity which is 50 times greater than the thermal conductivity of water. On the one hand this is good because it smooths out the temperature and inhibits thermal stratification. On the other hand it makes it necessary to place the metal in the depth of the Dewar flask without reaching the limits of the thermal protection column which poses a definite inconvenience.

In our case the development of an operating thermostat was preceded by investigations of the phase transformation process (fusion-solidification) of gallium and its alloys using small (10 g) specimens primarily for the purpose of finding a method to counteract supercooling. The initial components consisted of technical gallium from the Volkhovsk aluminum plant and of GPL refined gallium from the State Scientific Research and Design Institute of the Rare Metal Industry. These metals and their eutectic alloys with tin, zinc, indium and aluminum, which exhibited excessive supercooling effects (10° and more in the absence of shaking) were alloyed with various elements. A total of 87 alloys was prepared and investigated. In addition to alloying, we tried a small deviation of the alloy composition from the eutectic ratio and we also tried treating the metal with ultrasound (for the purpose of introducing insoluble additives); these two last measures turned out to be ineffective. In regard to alloying, it was established that the introduction of Cd, Na, Pb, Sb, Si largely supports supercooling effects (the interval δt of supercooling is increased, and the stability of the supercooled state is increased); the introduction of Ce, ^{/152}Cu, Mo produces a small decrease in the supercooling interval (2-6°); the introduction of Bi, Li substantially decreases the supercooling of some alloys. Our results are not exhaustive but so far we have found two alloys which may find practical applications. These consist of the eutectic Ga-Zn (95 percent Ga, 5 percent Zn, $t_{\text{fusion}}=25^\circ$), alloyed with lithium (2-3 percent Li) and with bis-

muth (0.05-0.1 percent Bi), as well as the eutectic Ga-Sn (92 percent Ga, 8 percent Sn, $t_{\text{fusion}}=20^\circ$) alloyed with bismuth (0.5-1 percent Bi) and lithium (0.5-1 percent Li). The latter alloy is not perfected; it is quite removed from a eutectic composition so that the phase transition on the temperature graph corresponds to an incomplete stoppage of temperature.

In general the purity of the initial metal, the accuracy of the eutectic ratio between components, and the absence, as much as possible, of impurities

which do not enter into the given eutectic composition, are of prime significance. Thus in our experiments, observations showed that while the process of phase transformation took place the temperature of pure gallium or its eutectic did not even vary by 0.1° whereas in the case of technical gallium under identical conditions the temperature difference at the beginning and end of the transformation reached a value of $2-3^\circ$.

We built a thermostat having the form of a double-walled polyethylene case which was placed on the housing of the gravity meter with both units placed in the Dewar flask. The space between the walls of the case contained a metal with a low melting point (fig. 2). We used the gallium-tin eutectic with an addition of bismuth and lithium. Although this alloy has not been perfected, it does operate at the required temperature (20°). Unfortunately the gallium was not pure but was of a cheaper technical variety which, of course, weakened the thermal stabilization effect. Nevertheless, 500 g of the alloy provided for a decrease in the internal temperature variation to $0.04^\circ/\text{hr}$ when there was a 20° temperature drop for a period of 10 hr. When gallium is absent, the temperature of the gravity meter under the same conditions varies from $2-3^\circ/\text{hr}$. The thermostat compensates for thermal shocks in a satisfactory manner. The transportation of the device from a temperature of 5° below zero to the street where the temperature was 40° , produced only a 0.06° temperature rise in the thermal chamber during the first half-hour. During the second half-hour the temperature rose by 0.03° more. However, the investigation was not completed. The effect of the thermostat on the accuracy of the gravity meter was not verified. /153

In planning future investigations we should provide for the testing of a large number of elements (magnesium first) for the purpose of finding those alloying additives which effectively inhibit supercooling as well as to determine the composition of the tetradic eutectic gallium-zinc-aluminum-silver which is interesting from the point of view of obtaining an alloy with a relatively large specific heat of fusion. The obvious prerequisite for such investigations is the fact that the above elements do not enter into a chemical reaction with each other and consequently when they are mixed, there is an additive effect in regard to the specific heat of fusion of the initial components (gallium 10 cal/g, zinc 25 cal/g, silver 25 cal/g, aluminum 90 cal/g).

Thermostat with Hydrocarbons

Hydrocarbons have a specific heat of fusion per unit weight which is greater than for gallium but are not as good in regard to heat of fusion per unit volume. The special feature of hydrocarbons is their low thermal conductivity so that a column of material poured into the Dewar flask produces thermal insulation which is as good as that provided by the best insulators, such as cotton and wool.

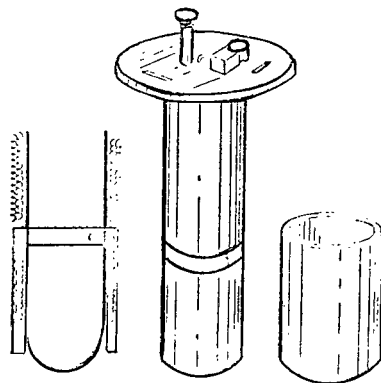


Figure 2. The internal part of the gravity meter and the case (with the thermostatic material) which fits on it.

First of all we investigated a heavy fraction which was obtained during the production of diesel fuel. The cooling and heating of one liter of this material in the Dewar flask exhibited a definite thermal effect. During cooling the thermal curve had a clearly defined area at 16°C. During heating the effect was less pronounced and the area was not obtained. We were unable to find the reason for this. Possibly it is due to a much greater temperature drop during heating, (the temperature in the thermal box was about 70°) than during cooling (from 0 to -10° on the street). In addition to the mode where there was a sharp change in heat and cold, the thermostat was also tested under conditions of natural temperature fluctuations in the street. For purposes of comparison, water in other Dewar flasks was tested simultaneously. Then the Dewar flasks were exchanged and the water was poured into the flask previously containing the hydrocarbon while the hydrocarbon was poured into the flask previously occupied by the water. The results of the test do not show an appreciable advantage of using diesel fuel in place of water. The reason for this is that, apparently, the particular diesel fuel which was used is not a pure substance but rather a mixture of components with different properties. Only a small part of these "operate" while transforming from a solid state to the liquid state and vice versa. The remaining components are always in the same state and merely serve as a ballast. This again shows that it is desirable to utilize pure substances.

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We turned to saturated hydrocarbons of the normal series (see table) in view of their availability (the other isomers are too exotic). Their density ranges from 0.70-0.77 g/cm and their heat of fusion per unit volume is from 15 to 22

cal/cm³. The thermal properties of these hydrocarbons are shown in the table (ref. 2).

The results of experiments with these substances turned out quite well. One liter of heptadecane in the Dewar flask maintained a temperature within the limits from 20-21.5°C for a period of 5-7 days and more under conditions of natural outside temperature variations by 6-8° with respect to the given temperature. The short term but sharp temperature fluctuations were completely removed. These occurred sometime during the 24-hour period in a controlled experiment with water (fig. 3a). Figure 3b shows the graph of the internal temperature of the gravity meter which was thermostatically controlled with heptadecane under conditions of forced alternations of heat and cold. For comparison purposes a gravity meter with a simple Dewar flask, with two flasks placed one inside the other and also with a water-filled flask were investigated simultaneously. As we can see, all of these other methods are much less effective.

TABLE

Formula	Name	Fusion temperature, degrees	Specific heat of fusion
C ₁₂ H ₂₆	n-dodecane	-10	20-30 cal/hr
C ₁₃ H ₂₈	n-tridecane	-6	
C ₁₄ H ₃₀	n-tetradecane	+5.5	Data on the specific heat of fusion are incomplete.
C ₁₅ H ₃₂	n-pentadecane	+10	
C ₁₆ H ₃₄	n-hexadecane	+16	
C ₁₇ H ₃₆	n-heptadecane	+22	
C ₁₈ H ₃₈	n-octadecane	+28	

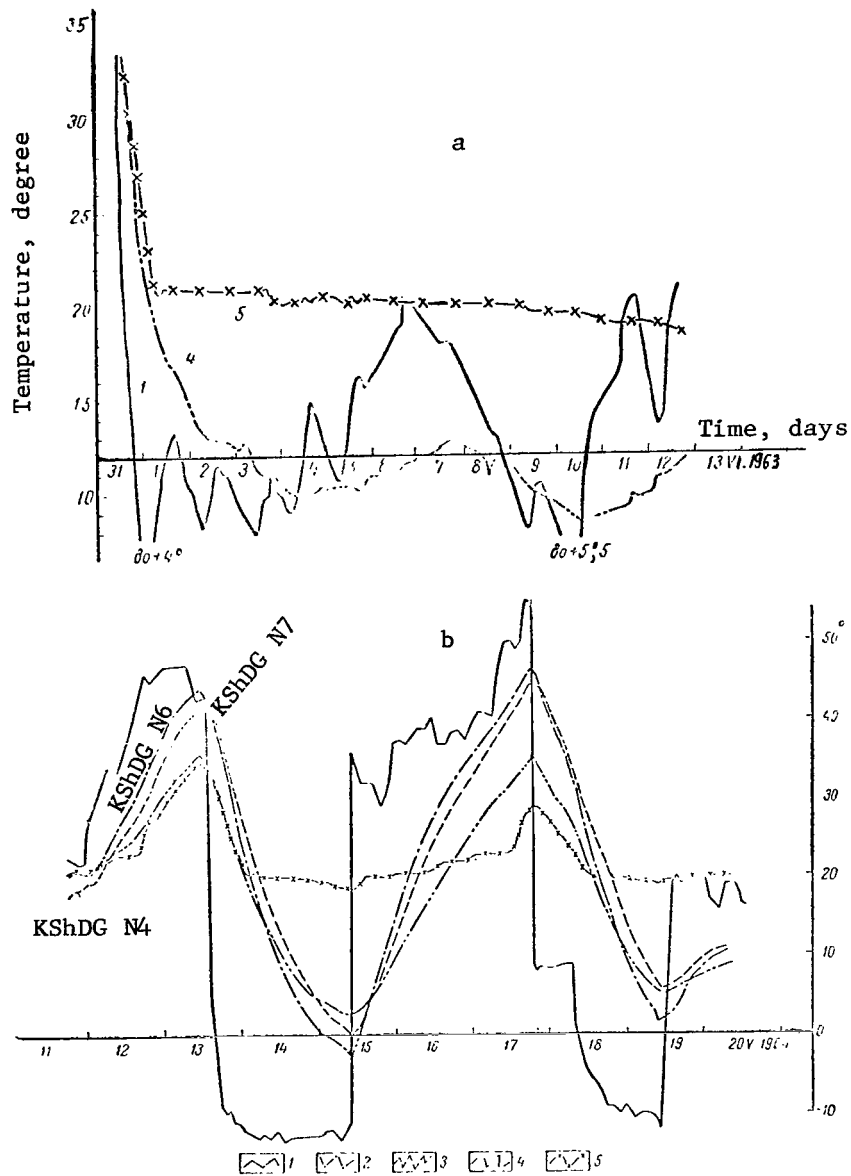


Figure 3. Test curves for different versions of the thermostat: a, under conditions of natural external temperature fluctuations; b, under conditions of forced temperature fluctuations; 1, external temperature; 2, the temperature of a conventional gravity meter; 3, the temperature of a gravity meter with a double Dewar flask; 4, the temperature of the gravity meter with a water jacket; 5, the temperature of the gravity meter with a heptadecane jacket.

The gravity meter with a disconnected temperature compensator ("gravity meter-thermometer"), which was thermostatically controlled with heptadecane filler, in the course of very rapid experiments, exhibited a zero drift of 1-3 milligals/hr which is 10-20 times less than the drift in the same gravity meter without a compensator and without heptadecane filler, although it substantially exceeds this quantity for a conventional gravity meter with a compensator and without heptadecane (0.1 milligals/hr).

At the present time the question is posed concerning the fabrication of a case which would make it possible to fill the space above the quartz system, inside the connecting vessel, with an active substance right up to the external panel so that hermetic insulation will exist only in the narrow channels of the illuminator, microscope, microlead screw, range switch and of the thermometer.

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